(CZ0400049)

IAPP RUS'O PCT/PTO 01 MAR 2006

Amended Claims under PCT Article 34

- 1. A method of preparing the dichloropropanols 1,3-dichloro-2-propanol and 2,3-dichloro-1-propanol by hydrochlorination of glycerine and/or monochloropropanediols with gaseous hydrogen chloride with catalysis of a carboxylic acid, characterized in that said hydrochlorination is carried out solvent-free in at least one continuous reaction zone at reaction temperatures in the range of 70-140 °C and with continuous removing of the water of reaction by distillation at reduced pressure, the liquid feed containing at least 50 % by weight of glycerine and/or monochloropropanediols.
- 2. The method according to claim 1, characterized in that the liquid feed contains 80-100 % by weight of glycerine.
- 3. The method according to claim 1, characterized in that the liquid feed contains, as the monochloropropanediols, 3-chloro-1,2-propanediol and/or 2-chloro-1,3-propanediol.
- 4. The method according to any of the preceding claims, characterized in that the catalysis is made with acetic acid.
- 5. The method according to any of the preceding claims, characterized in that the reaction is carried out at a temperature of 100-110 °C.
- 6. The method according to any of the preceding claims, characterized in that the distillation at reduced pressure is carried out in a rectification zone linked to the reaction zone.
- 7. The method according to claim 6, characterized in that, together with the removing of the water of reaction by distillation, at least partial primary collection of the product dichloropropanols is made.
- 8. The method according to claim 6 or 7, characterized in that secondary collection is made, from which dichloropropanols and monochloropropanediols are recycled to the process.
- 9. The method according to claim 8, characterized in that the secondarily collected residual balance of the reaction mixture is subjected to distillation under reduced pressure in order to separate the higher boiling waste products as the distillation

BEST AVAILABLE COPY

2

residue and the dichloropropanols and monochloropropanediols, recycled to the reactor, as the distillate.

- 10. The method according to any of claims 1-5, characterized in that it is carried out in a cascade of continuous flow reaction zones wherein the water of reaction is collected, together with partial collection of the product dichloropropanols, by distillation at reduced pressure, located always downstream the individual reaction zones of the cascade, and the distillation residue is fed into the next zone of the cascade.
- 11. The method according to claim 10, characterized in that the reaction mixture exiting from the last step of the cascade is subjected to a two-step distillation, wherein in the first step the water of reaction is separated together with the dichloropropanol reaction product as the distillate and in the second step the higher boiling waste products are the distillation residue and the dichloropropanols separated monochloropropanediols are separated as the distillate and are recycled back to the process, preferably into the first step of the cascade.
- 12. An apparatus for carrying out the method of any of claims 1-9, wherein the apparatus comprises a circulation reactor, consisting of a vertical cylinder, with external circulation, in which there is located a vacuum rectification column downstream of the reactor, for continuous returning the distillation residue from the vacuum rectification column back to the reactor and for continuous collection of a mixture of the dichloropropanol product, the reaction water and the residual hydrogen chloride as a distillate.
- 13. The apparatus according to claim 12, wherein the apparatus further comprises a vacuum distillation device for continuous removing of the undesired high-boiling waste products as distillation residue and back to the reactor recycled dichloropropagols and monochloropropagediols as distillate, located downstream the vacuum rectification column.
- 14. An apparatus for carrying out the method of claims 10 or 11, wherein the apparatus comprises a cascade of continuous flow reactors, in which there are vacuum distillation devices located downstream the individual steps of the cascade for distilling off the water of reaction and a part of the dichloropropanol product; the distillation residue being led to the subsequent member of the cascade.

Empf.zeit:27/06/2005 17:02

3

- 15. The apparatus of claim 14, wherein the number of the members of the cascade is 1 to 5.
- 16. The apparatus of claim 14 or 15, wherein the number of the members of the cascade is 3.

TEST AVAILABLE COPY